



Standard Test Methods for Ashing Cellulose¹

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^{ε1} NOTE—Keywords and unit of measurement statement were added editorially in June 1995.

1. Scope

1.1 These test methods cover four ashing methods for cellulose. These are intended for use on unbleached and bleached cellulose in sheeted or bulk fiber form. Each one of the test methods has advantages, so that preference applications exist for all four.

1.2 The test methods appear as follows:

Test Method A—Ash in Cellulose at 575°C	Sections 6 to 11
Test Method B—Sulfated Ash in Cellulose at 575°C	12 to 17
Test Method C—Ashing Cellulose by Schoniger Oxidation	18 to 22
Test Method D—Wet Ashing of Cellulose for Inorganics	23 to 29

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Note 12.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1348 Test Methods for Moisture in Cellulose³

3. Summary of Test Methods

3.1 *Test Method A, Ash in Cellulose at 575°C*—This test method measures the ash content of cellulose, which is defined for this test as the residue remaining after ignition at 575 ± 25°C until all carbon has been burned off. It is the simplest of the four test methods for the determination of ash content only, and it should not be considered as a standard preparative procedure for elemental analysis.

NOTE 1—The ash content at this ignition temperature is a reasonable measure of the mineral salts and inorganic foreign matter in the cellulose.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 06.03.

The weight of ash obtained varies with the temperature of ignition. Higher temperatures (850°C) will convert calcium carbonate and other carbonates to the oxides and thus give lower values for ash. The composition of ash may vary with the pulping process employed for manufacture, which limits the significance of the ash determination in absolute terms.

3.2 *Test Method B, Sulfated Ash in Cellulose at 575°C*—In this ashing procedure the metal salts are converted to sulfates by treatment with sulfuric acid and ignition at 575 ± 25°C to overcome limitations of Test Method A. It should not be considered as a standard preparative test method for elemental analysis.

NOTE 2—Conventional dry ashing at high temperature (over 850°C) results in loss of carbon dioxide from metal carbonates, decomposition of magnesium sulfate, and loss of sodium salts. Ashing at lower temperatures (575°C) gives a better estimate of the inorganic impurities, but has the disadvantage of requiring a long ignition time and also the heat produced by the burning pulp can raise the temperature far above 575°C for a short period of time, converting calcium carbonate to the oxide in varying amounts. The sulfated ash method overcomes these objectionable features and has additional advantages. It is relatively insensitive to ignition time. Ash weight remains constant during weighing, and because the ash does not melt it will not attack the crucible.

3.3 *Test Method C, Ashing Cellulose by Schoniger Oxidation*—This test method is generally applicable to ashing cellulose for subsequent analysis of inorganic constituents. The procedure utilizes the Schoniger technique in which the cellulose is burned in a combustion flask and all combustion products are dissolved in an aqueous medium, thus avoiding the possibility of physical loss of sample such as can occur in dry ashing (Test Methods A and B). A limitation is the relatively small sample of 1 g, which can be ashed without excessively large apparatus in which to carry out the oxidation step. Small specimens may not be adequately representative where a particulate contaminate is involved.

3.4 *Test Method D, Wet Ashing of Cellulose for Inorganics*—This test method is recommended for ashing cellulose for subsequent analysis of inorganic constituents. The procedure employs saturation of the cellulose with a concentrated solution of hydrogen peroxide followed by incremental addition to a small volume of concentrated sulfuric acid. It has the advantage over Test Method C of not requiring special apparatus and can be readily applied to samples of 5 to 10 g. The test method becomes tedious and less convenient for use where larger specimens such as 50 or 100 g are required.